CORROSION OF VANADIUM BY MOLTON BISMUTH

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By

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ABSTRACT

In this study vanadium was evaluated as a container material for molten bismuth in a liquid-metal-cooled system. A rough appraisal of vanadium as a container material for sodium was also included.

The investigation took two forms. The first series of tests utilized crucibles of vanadium containing bismuth. Results were determined in terms of three independent variables, viz: temperature, time, and degree of agitation. Dependent variables were (1) solubility of vanadium in bismuth and (2), depth and type of corrosive penetration.

The second series of tests utilized tubes of vanadium containing a small amount of bismuth. The tubes were tilted at five minute intervals while the ends of the tubes were maintained at constant but widely separated temperatures. Deposits in the low temperature end furnished physical evidence of thermal gradient mass transport.

Results demonstrated that vanadium

1. was susceptible to mechanical (corrosion-erosion) attack by bismuth between

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temperatures of 550° C and 700° C,

2. had high solubility in bismuth,
3. suffered severe intergranular corrosive attack at temperatures above 900° C
4. demonstrated a strong propensity for thermal gradient mass transport,
5. was attacked, during the mass transfer experiments, at a particularly rapid rate in the high temperature region because of the cyclic removal of dissolved vanadium in the low temperature region.

In the included experiments with molten sodium, the vanadium showed little resistance to attack.

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I SUMMARY

The purpose of this study was to evaluate vanadium as a container material for molten bismuth in a liquid-metal-cooled system. A rough appraisal of vanadium as a container material for sodium was also included.

The investigation took two form. The first series of tests utilized crucibles of vanadium containing bismuth. Results were determined in terms of three independent variables, viz.: temperature, time, and degree of agitation. Dependent variables were (1) solubility of vanadium in bismuth, determined by chemical analysis, and (2), depth and type of corrosive penetration, determined by metallographic inspection.

The second series of tests utilized tubes of vanadium containing a small amount of bismuth. The tubes were tilted at five minute intervals while the ends of the tubes were maintained at constant but widely separated temperatures. Deposits in the low temperature end furnished physical evidence of thermal gradient mass transport. Cold-end deposits were analyzed chemically. Identification of deposits were checked by X-ray spectrographic analysis. Identification of the form taken by the deposit was made by metallographic inspection. Corrosive attack of

the high temperature end of the tube was also studied metallographically.

Results were unfavorable. The vanadium:

1. was susceptible to mechanical (corrosion-erosion) attack by bismuth between temperatures of 550°C and 700°C,

2. had high solubility in bismuth, solubility varying almost linearly with temperature,

3. suffered severe intergranular corrosive attack at temperatures of 900°C and above,

4. demonstrated a strong propensity for thermal gradient mass transport,

5. was attacked, during the mass transfer experiments, at a particularly rapid rate in the high temperature region because of the cyclic removal of dissolved vanadium in the low temperature region.

In the included experiments with molten sodium, the vanadium showed little resistance to attack.

II INTRODUCTION

A. Background

Molten bismuth has many properties which make it a highly desirable coolant in nuclear reactors. It has low neutron absorption cross-section, a high transport cross-section, and excellent alloying characteristics with uranium. It has a relatively low melting point (268°C) combined with a high boiling point (1460°C). Although its specific heat is quite low, its high density gives it good heat transfer characteristics on a volumetric basis. Efforts to utilize its advantages in a liquid-metal-cooled or a liquid-metal-fueled reactor have launched a search for a good container material for molten bismuth. Such a material must, among other things:

(1) not be corroded by the molten bismuth,

(2) have favorable microscopic nuclear characteristics

(3) resist mass transport in circulation loops involving temperature cycling.

(4) have suitable high-temperature strength and creep resistance,

(5) be reasonably easy to fabricate,

Vanadium, when considered as a possible container material for molten bismuth, has received rather cursory but generally favorable mention (1). Preliminary static tests used to screen out obviously unsuitable materials indicate that vanadium resists corrosion by molten bismuth. More exhaustive tests on those materials showing promise have naturally been focused first upon the ones which were most attractive from the stand-points of economy and ease of fabrication.

B. Scope

Investigations of the corrosion of vanadium by bismuth, and thermal gradient mass transport in a vanadium system involving thermal cycling of molten bismuth, were established as the primary aims of this study.

1. Corrosion studies

Corrosion of a solid metal by a molten metal is minimal when either the free energy of the reaction is positive, or when an intermetallic film or protective layer is formed through which the diffusion rates are small (3). It was assumed, as a working hypothesis, that some kind of intermetallic film would be formed in a vanadium-bismuth system, and the apparatus was designed in such a way as to subject this (assumed) film to varying degrees of agitation. Erosion of the film and of the inner surfaces of the container were expected to vary the results of the

corrosion tests. For purposes of comparison, containers of a 5% chromium - 1½% silicon steel were included in the runs. This steel, known as Croloy 5 Si, has been the subject of extensive tests at Brookhaven National Laboratory (2). In addition, at test temperatures below the boiling point of sodium, vanadium crucibles containing sodium were included for rough qualitative test of resistance of vanadium to attack by molten sodium.

Test and analysis procedures for this series of tests were designed to develop a broad range of information in a relatively short time rather than to achieve pin-point accuracy. They were principally intended to indicate the direction of any further investigation.

2. Thermal gradient mass transport studies

When solubility of the solid, or container, metal varies widely with changes of temperature, there is a probability that mass transfer will occur in a closed loop system involving physical circulation and simultaneous thermal cycling. Container metal, going into solution at the high temperature, precipitates out and forms a metallic growth or mass in the lower temperature region. Concomitantly, the removal of the dissolved component from the circulating material accelerates corrosion attack in the high temperature region (3). The tilt-tube process used in this investigation offers a simple and rapid test for mass

tranfer. The ends of a long tube of the container material are maintained at constant temperatures, one high, the other low, and the tube is tilted back and forth so that the contained molten metal is subjected to sudden and severe thermal cycling. Under these conditions, mass transport, when it occurs, may be expected to be severe and unmistakable.

C. Classification

For simplicity of handling and dissemination it was decided, upon advice of the thesis supervisor, to keep this report unclassified. Because of its narrow scope and in the light of similar reports in the literature, this study is clearly of an unclassified nature; however, information pertinent to the testing procedures and previous work done on allied subjects is often contained in compilations or in combination with classified material. Quotations from classified literature, either directly or indirectly, is therefore impossible and has been rigorously avoided. A list of classified literature containing pertinent material is included in the bibliography but not referred to in the text.

III PROCEDURE

A. Corrosion Tests

1. Apparatus

An electric furnace, eighteen inches in overall length, was wound and energized in such a way that isothermal conditions could be maintained over a fourteen inch length of its interior. Details of furnace construction are contained in Appendix A. The furnace was mounted on a teeter-board tilting 20° from the horizontal in each direction on bearings located in line with the center of the furnace. A motor-driven crank arm lifted the weighted teeter-board through the horizontal (the point of unstable equilibrium) from where it dropped freely the remaining 20° onto a spring which bounced it several times. This action was repeated twelve times per minute. The purpose was to subject specimens placed in the center of the furnace to a mild form of agitation, little more than a tilting action, whereas specimens located away from the center received repeated jolting, progressively increasing in violence as the ends of the furnace are approached. Figure 1 is a diagram of the furnace mechanism.

2. Specimens

Specimens were small cylindrical crucibles of vanadium and of Croly 5 Si type steel. Interior dimensions were 0.25 inches in diameter by 1.00 inch in length.

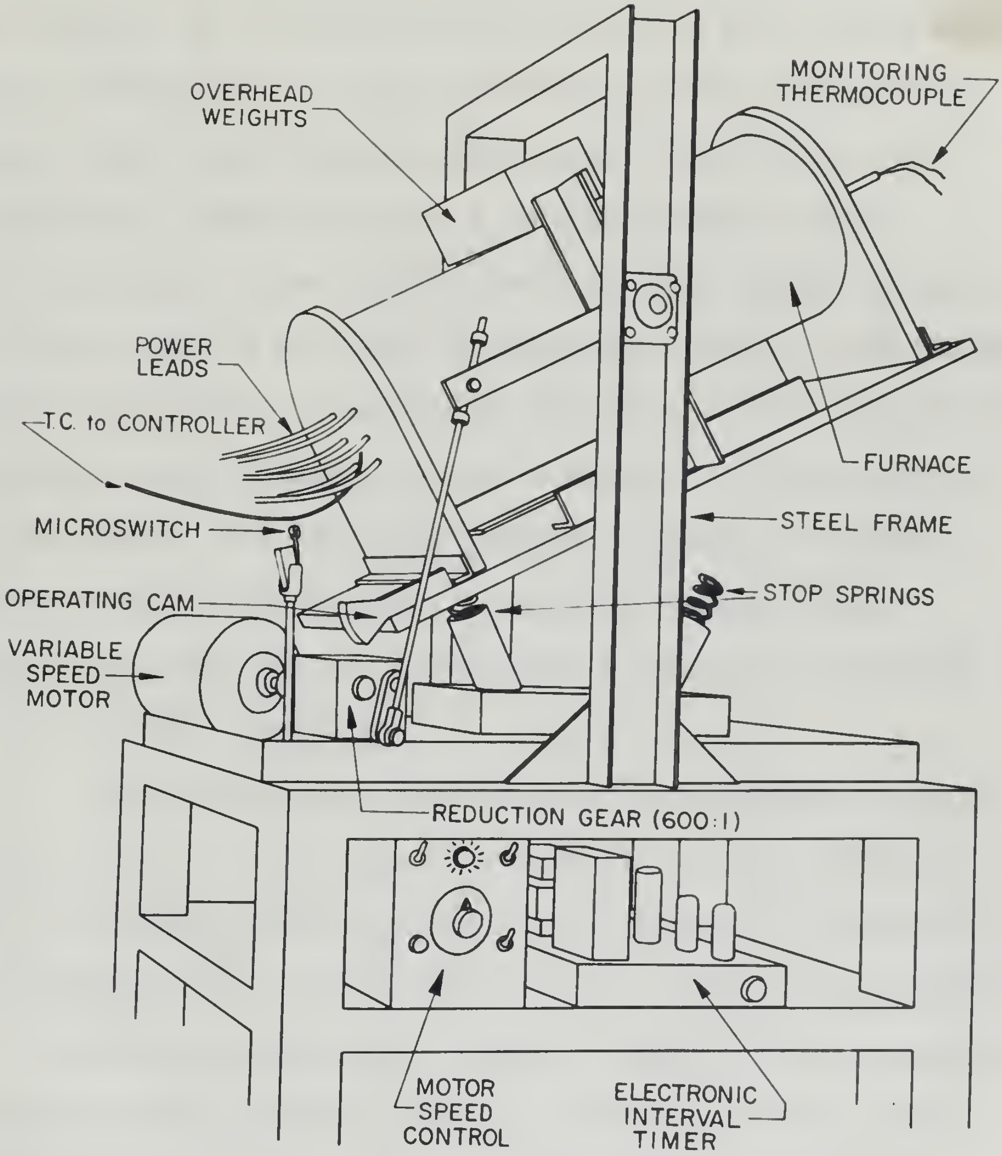


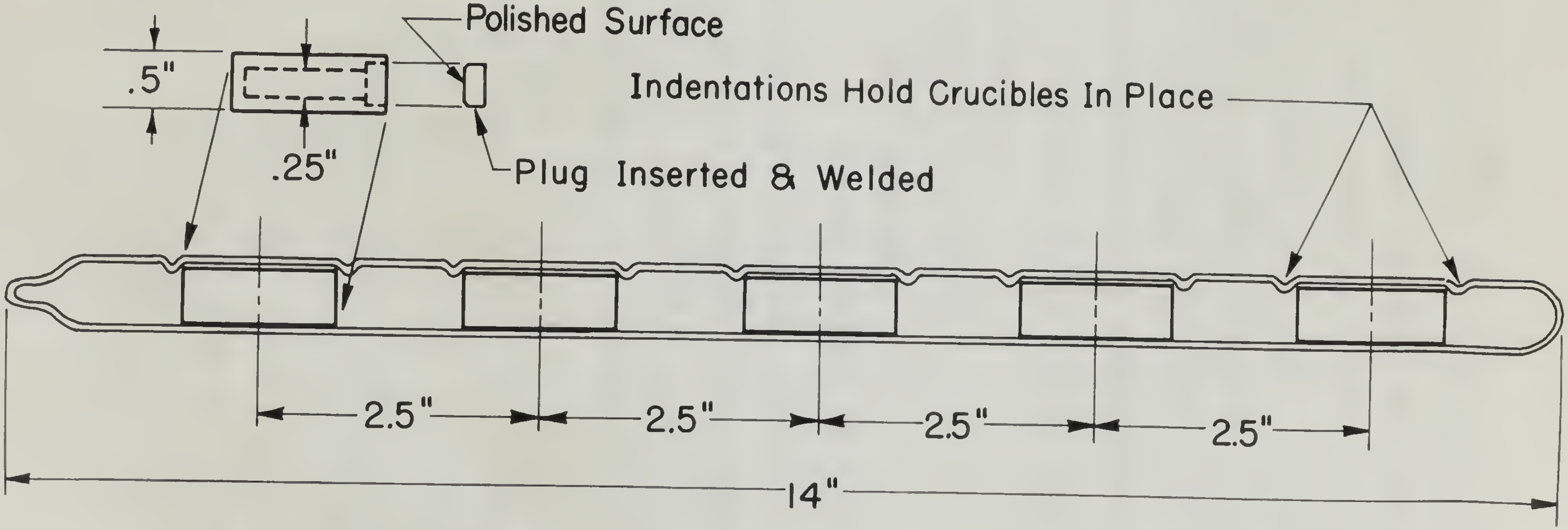
FIGURE 1 FURNACE APPARATUS

Crucible construction and dimension are shown in Figure 2. A few of the crucibles were filled about one half full (approximately 4.5 gram) with purified bismuth. The inner surface of the cap was polished for later metallographic examination. Inner surfaces of the rest of the crucible were reamed smooth and pickled in nitric acid. The crucibles were placed in a tight-fitting copper jacket which was water cooled and the caps welded on with an electric arc in an argon atmosphere using a water-cooled tungsten electrode. Cooling of the body of the crucible during welding was dictated by the necessity of completing the weld before the contained metal (sodium or bismuth) reached boiling temperature. Crucible fabrication, loading, and welding are discussed in detail in Appendix B.

3. Testing procedures

The crucibles were wrapped in tantalum foil and placed, five to a tube, in Vycor glass tubing, fourteen inches in length overall, which was subsequently evacuated and sealed off. Depressions in the glass tube maintained the crucibles rigidly in place. Crucibles were mounted 2½ inches apart at their centers, overall crucible displacement being 12 inches, end to end. Mounting of crucibles is shown in Figure 2. The center position in each tube was occupied by a vanadium crucible containing bismuth which received mild agitation. The two end

Figure 2 Corrosion Test Specimens



V-Crucibles, Each Containing 4 Grams of Bi

positions, subjected to the most severe agitation were occupied by a vanadium crucible and steel crucible, respectively, both containing bismuth. Intermediate position were not filled in all runs. When filled, they were occupied by vanadium crucibles, one containing bismuth the other containing sodium. In terms of the t three independent variables intruduced in these tests, crucibles were placed in the furnace according to the schedule of Table 1.

4. Analysis procedures

(a) Chemical

Upon removal from the furnace, crucibles were set vertically, without cooling, for a short time so that particles eroded from the inside of the crucibles had opportunity to settle at the end, then quenched rapidly in cold water. It was expected that the crucible metal taken into solution by the bismuth might precipitate out upon quenching but that freezing would occur so rapidly that this precipitate would remain in suspension. The cap, bearing the prepared surface, was then cut off and prepared for metallograpic inspection. Those crucibles containing bismuth were chucked in a lathe and the crucible and outer surface of the bismuth turned off. The remaining bismuth was cleaned by dissolving to about 2/3 of

Temperature (degrees C)	Mild Agitation	Intermediate Agitation	Severe Agitation
550	24 hours	24 hours	24 hours
550	96 "	96 "	96 "
550	218 "	218 "	218 "
550	360 "	360 "	360 "
630	96 hours		96 hours
720	96 hours		96 hours
800	24 hours	24 hours	24 hours
800	48 "	48 "	48 "
800	96 "	96 "	96 "
800	192 "	192 "	192 "
800	338 "	338 "	338 "
900	96 hours		96 hours
1000	24 hours		24 hours
1000	72 "		72 "
1000	144 "		144 "

TABLE I (a) Schedule of test periods for vanadium crucibles containing bismuth

Temperature (degrees C)	Mild Agitation	Intermediate Agitation	Severe Agitation
550			24 hours
550			96 "
550			216 "
550			360 "
630			96 hours
720			96 hours
800			24 hours
800			48 hours
800			96 hours
800			192 hours
800			338 hours
900			96 hours
1000			24 hours
1000			72 "
1000			144 "

TABLE I (b) Schedule of test periods for steel crucibles containing bismuth

Temperature (degrees C)	Mild Agitation	Intermediate Agitation	Severe Agitation
550		24 hours	
550		96 "	
550		218 "	
550		360 "	
800		24 hours	
800		48 "	
800		96 "	
800		192 "	
800		338 "	

TABLE I (c) Schedule of test periods for vanadium crucibles containing sodium

former size in nitric acid. This was then divided into two two parts and the parts individually analyzed chemically as a check for homogeneity. Analyses of the two parts are listed separately in Table II. The standard determination for vanadium (potassium perchlorate-sulfuric acid) could not be utilized because of interference with the test by the bismuth, and use of a considerably less accurate colorimetric test for vanadium was enforced. Appendix C details chemical analysis procedures utilized.

Crucibles containing sodium were cut open and immersed in distilled water. After the reaction had subsided, the crucible was removed and its interior was flushed with distilled water, the washings being added to the solution. The sodium content was determined from analysis of the sodium hydroxide formed. All solid particles remaining were taken into solution with acid and the resulting solution tested for amount of vanadium present.

(b) Metallographic

The caps were sectioned perpendicularly to the prepared surface, mounted in plastic and polished. Without etching, the surfaces were examined by means of a metallograph. To prevent personal bias or predilection, an independent observer, not appraised of the specimen test conditions, examined the entire cross section of the surface exposed to corrosive action.

The observer then selected a representative section typifying the conditions of the surface, and this section was photographed.

B. Mass transfer tests

1. Apparatus

For this set of tests the previously described furnace was energized in such a way as to provide a localized point of high temperature near its center. An electronic interval timer was used to interrupt motor action so that the furnace was tilted down on the alternate end every five minutes. The bouncing action described in previous section was damped out. Appendix F discusses the switch and timer action for cycle control in the tilt-tube mass transfer tests.

Two large cylindrical blocks of stainless steel were fabricated to fit over 1½ inches of each end of the tilt-tube specimens. These blocks acted as heat reservoirs and were used to stabilize the temperatures of the ends of the tilt-tubes. Thermocouples inserted in the blocks were used to monitor tilt-tube end temperatures. The tilt-tube heat-reservoir-blocks assembly is shown in Figure 3.

One block was maintained at 900°C. The other block stabilized at 390°C when three tilt-tubes were in operation simultaneously, at 320°C when two tubes were in operation,

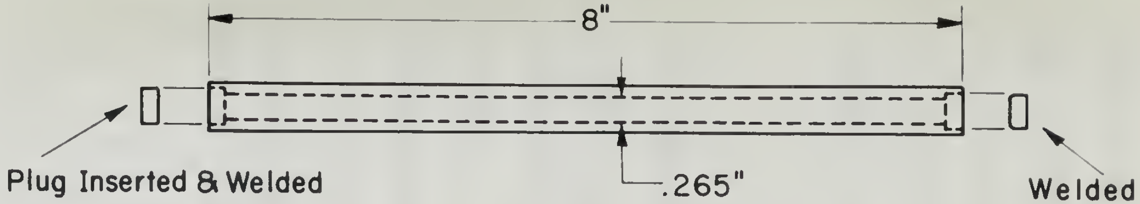
and was held at 300°C (by addition of external heat) when only one tube was left.

2. Specimens

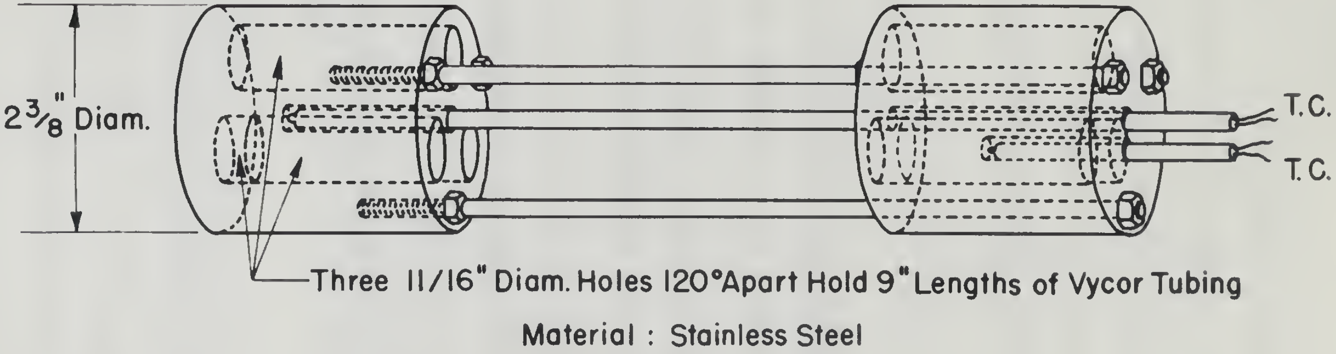
Specimens were eight inch long tubes of vanadium drilled from extruded rod as discussed in Appendix D. A tilt-tube for the mass transfer test is depicted in Figure 3. Caps with polished inner surfaces, were welded over the ends. Contents were ten grams of bismuth. The vanadium tubes were wrapped with tantalum foil and placed in Vycor glass tubes, evacuated, and sealed. The wrapped vanadium specimens were made to fit tightly in the glass tubes in order to attain good heat transfer.

3. Analysis

At selected time intervals the tilt-tube assembly was opened and a tube removed. The tube was quenched vertically with the high temperature end down. Ends of the tube were cut off and sectioned. One half of the low temperature end was polished for X-ray spectographic analysis and for retention as physical evidence of mass transfer. The deposit in the other half was carefully chipped out and used for chemical analysis. The high-temperature end was sectioned and polished for metallographic examination.



Mass-Transfer Tilt Tube Containing 10 g. of Bi



Heat Storage Blocks Used In Mass-Transfer Tilt Tube Experiment

Figure 3 Mass Transfer Test Specimens

IV RESULTS

A. Corrosion tests - vanadium by bismuth

1. Chemical analysis

Results of chemical analyses of the bismuth are tabulated in Table II. The mean solubility of vanadium in bismuth for each temperature at which the test was run is plotted in Figure 4. Mean solubility of steel (iron in solution) in bismuth is superimposed for purposes of comparison.

2. Metallographic analysis

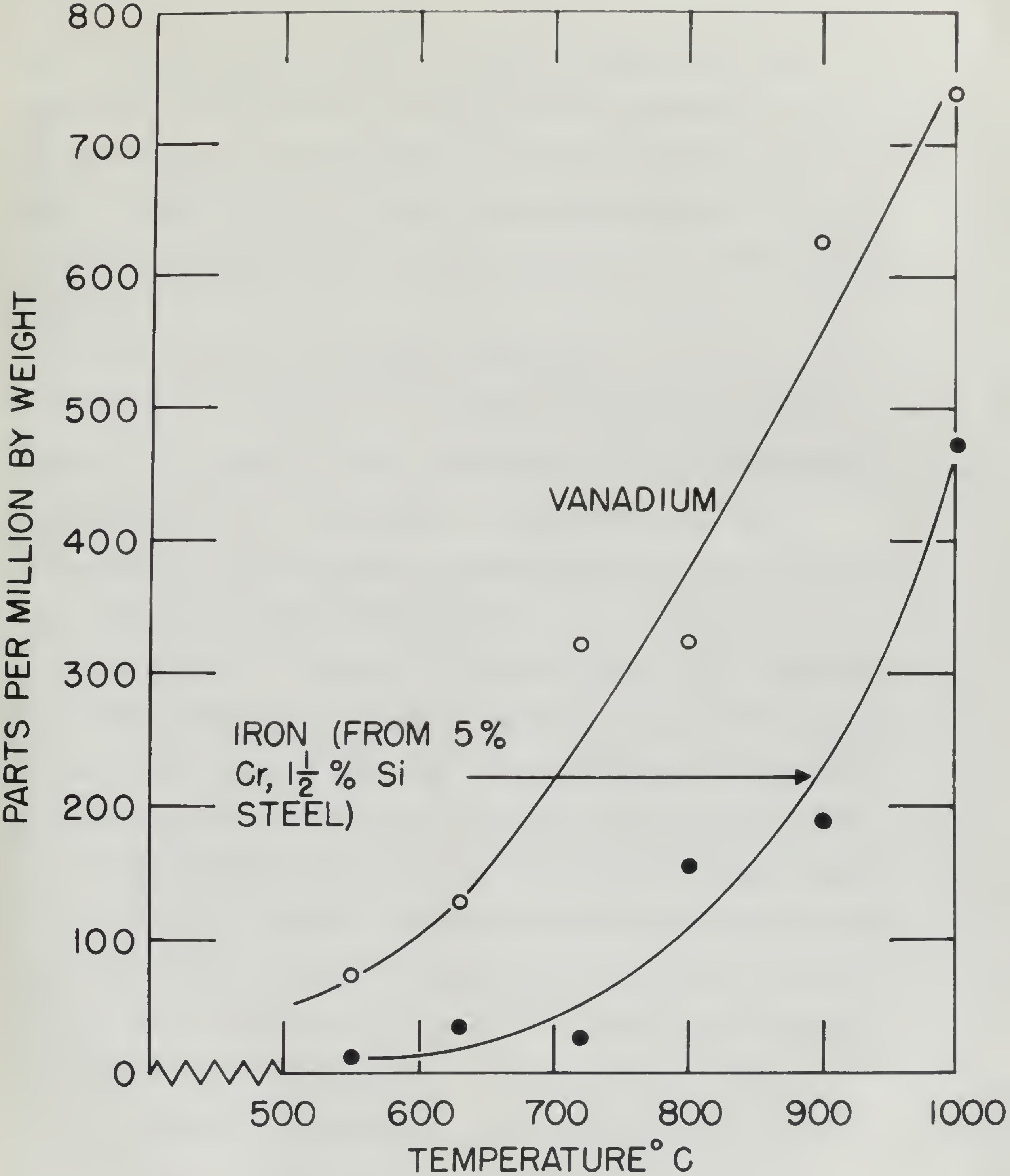
Neither degree of agitation or period in the furnace (shortest period - 24 hours) had significant effect upon final solubility, but did have a marked effect upon the appearance of the attack on the polished interior surface. Figures 5 to 13 are photomicrographs of polished vanadium surfaces following exposure to molten bismuth for the longest and shortest test periods at all test temperatures. Both mild and severe agitation are represented in these photomicrographs.

3. Crucible failure

Five crucibles failed under test. Three of these, exposed for 96, 192, and 336 hours, respectively, were at 800° C. All three occupied end positions in the furnace where agitation was most severe. The other two failures occurred at 1000° C when exposed for 144 hours.

CRUCIBLE MATERIAL	LIQUID METAL CONTAINED	TEMPERATURE Degrees C	DURATION OF TEST (Hours)	DEGREE OF AGITATION	CHEMICAL ANALYSIS Crucible Material in Liquid Metal (ppm)		TYPE OF ATTACK	DEGREE OF PENETRATION inches x10^{-3}
Vanadium	Bismuth	550	4	Mild	30	30		
Vanadium	Bismuth	550	96	Mild	30	30		
Vanadium	Bismuth	550	218	Mild	220	220		
Vanadium	Bismuth	550	360	Mild	210	220		
Vanadium	Bismuth	550	24	Intermediate	30	30		
Vanadium	Bismuth	550	96	Intermediate	30	30		
Vanadium	Bismuth	550	218	Intermediate	[illegible]	310		
Vanadium	Bismuth	550	360	Intermediate	30	30		
Vanadium	Bismuth	550	24	Severe	30	30	Erosion	0.5
Vanadium	Bismuth	550	96	Severe	30	30	pits	
Vanadium	Bismuth	550	218	Severe	140	140		
Vanadium	Bismuth	550	360	Severe	30	30	Erosion pits	.75
Vanadium	Bismuth	630	96	Mild	150	150	Erosion plus	
Vanadium	Bismuth	630	96	Severe	110	110	intergranular	1.8
Vanadium	Bismuth	720	96	Mild	350	410	Intergranular	2.0
Vanadium	Bismuth	720	96	Severe	260	270	General surface	0
Vanadium	Bismuth	800	24	Mild	310	290	Erosion	.6
Vanadium	Bismuth	800	48	Mild	310	350	General surface	0
Vanadium	Bismuth	800	96	Mild	290	290		
Vanadium	Bismuth	800	192	Mild	370	370		
Vanadium	Bismuth	800	338	Mild	350	340		
Vanadium	Bismuth	800	24	Intermediate	140	130	General surface	0
Vanadium	Bismuth	800	48	Intermediate	360	410		
Vanadium	Bismuth	800	96	Intermediate	320	310		
Vanadium	Bismuth	800	192	Intermediate	320	270		
Vanadium	Bismuth	800	338	Intermediate	340	400	Small erosion	0.4
Vanadium	Bismuth	800	24	Severe	250	290	Erosion pits	0.4
Vanadium	Bismuth	800	48	Severe	460	500		
Vanadium	Bismuth	800	96	Severe	failed			
Vanadium	Bismuth	800	192	Severe	failed			
Vanadium	Bismuth	800	338	Severe	failed			
Vanadium	Bismuth	900	96	Mild	580	590	Intergranular	3.0
Vanadium	Bismuth	900	96	Severe	650-640	650-670	Intergranular	4.5
Vanadium	Bismuth	1000	24	Mild	900	980	Intergranular	1.8
Vanadium	Bismuth	1000	72	Mild	800-920	460-440	Intergranular	4.5
Vanadium	Bismuth	1000	144	Mild	failed			
Vanadium	Bismuth	1000	24	Severe	980	960	Intergranular	1.5
Vanadium	Bismuth	1000	72	Severe	650-350	870-560	Intergranular	2.3
Vanadium	Bismuth	1000	144	Severe	failed			
Croloy 5 Si	Bismuth	550	24	Severe	15		No attack	0
Croloy 5 Si	Bismuth	550	96	Severe	12			
Croloy 5 Si	Bismuth	550	218	Severe	11			
Croloy 5 Si	Bismuth	550	360	Severe	12			
Croloy 5 Si	Bismuth	630	96	Severe	32	37		
Croloy 5 Si	Bismuth	720	96	Severe	22	28		
Croloy 5 Si	Bismuth	800	24	Severe	38	37	Slight surface roughness	0.1
Croloy 5 Si	Bismuth	800	48	Severe	163	163		
Croloy 5 Si	Bismuth	800	96	Severe	235	237		
Croloy 5 Si	Bismuth	800	192	Severe	173	175		
Croloy 5 Si	Bismuth	800	338	Severe	173	172	Slight surface	0.1
Croloy 5 Si	Bismuth	900	96	Severe	188	---	roughness	0.75
Croloy 5 Si	Bismuth	1000	24	Severe	742	---	Subsurface voids	1.1
Croloy 5 Si	Bismuth	1000	72	Severe	305	---		
Croloy 5 Si	Bismuth	1000	144	Severe	846	---	Subsurface voids	3.0
Vanadium	Sodium	550	24	Intermediate	4300	---	Erosion pits	0.4
Vanadium	Sodium	550	96	Intermediate	16700	---		
Vanadium	Sodium	550	218	Intermediate	10900	---		
Vanadium	Sodium	550	360	Intermediate	197000	---	Erosion pits	0.4
Vanadium	Sodium	800	24	Intermediate	9500	---	General surface	0
Vanadium	Sodium	800	48	Intermediate	4900	---		
Vanadium	Sodium	800	96	Intermediate	6500	---		
Vanadium	Sodium	800	192	Intermediate	7300	---		
Vanadium	Sodium	800	338	Intermediate	6100	---	Erosion pits	0.4

TABLE II. Corrosion test data sheet



SOLUBILITY OF CRUCIBLE MATERIAL IN BISMUTH

Figure 4

One of these two occupied a central (least agitated) position. No intermediate-agitation specimens were included in the 1000° C runs. No steel crucibles failed under test. Steel crucibles always occupied positions of most severe agitation at all temperatures and for all periods tested.

4. Wetting of crucible surfaces

Wetting of the surface in liquid-metal cooling is considered very important to heat transfer by some authors (4). Although its importance, in the presence of pure material, is disputed, type and thoroughness of wetting were recorded.

Below 700° C wetting of the vanadium by the bismuth was intermittent, the cohered bismuth being in the form of small beads loosely adherent to the surface. At 700° C film formation was evident but still intermittent, the film differing in color from the two metals from which it was formed. Between 700°C and 800°C clean patches of vanadium still appeared occasionally through the film. Above 800°C wetting was thorough - a thick film forming which was quite inseparable from the crucible sides.

In contrast, wetting in the steel crucibles did not appear below temperatures of 800°C and did not exhibit a high degree of adherence to the surface even in the 1000°C specimens. Such wetting as there was in the steel crucibles was entirely different in nature from that in vanadium crucibles.

B. Corrosion test - Croloy 5 Si by bismuth

Results are included in Table II. Photomicrographs of crucible inner surface attack are collected in Figure 14. Steel surfaces resisted attack far better than did the vanadium at all temperatures. Corrosion was relatively minor until every high temperatures were reached. Above 900° C. the steel was attacked in a peculiar way. Bismuth penetrated through a small fissure in the surface, dissolving away material at the bottom of the fissure to form a pocket or sub-surface void.

C. Corrosion tests - vanadium by sodium

Results are included in Table II. Photomicrographs of crucible inner surface attack are collected in Figure 15. Agitation was intermediate in all cases. Although results of chemical analysis are listed as parts of vanadium per million parts, by weight of sodium, this is by no means intended to portray solubility. All vanadium removed from inner surfaces of the crucible, including entrained particles eroded from the surface as well as vanadium taken into solution by the sodium, was included in the measurement.

D. Mass transfer tests

1. Physical evidence

Photographs of deposited material in the cold end of the tilt-tubes for cycling periods of 168 hours, and 336 hours are shown in Figure 16. Brittleness and fragility of the deposit caused loss and breakage in attempts to polish

the sections in several instances. There was nothing whatever in the cold end of the tube cycled for only one hour.

Deposit in the cold end of the tube cycled for 336 hours formed a pocket which trapped some of the liquid metal. The entrapped bismuth is clearly seen between the side of the vanadium tube and the deposited material in Figure 16 (b). A photomicrograph of the identical region may be seen in Figure 20. A photomicrograph of the deposit in the 168 hour test is shown in Figure 19 and a macrophotograph of this deposit is included in Figure 16 (a). The high temperature ends of the tubes which were cycled for 1, 168, and 336 hours are shown in Figure 17.

2. Chemical analysis

Chemical analysis of the deposited material from the low temperature ends of the tilt tubes are listed in Table III. The composition of the phases and the relative amounts of each present are not revealed by chemical analysis. The deposited material was loosely bonded to the tube walls and easily broken free.

3. Metallographic examination

Figure 18 is a collection of photomicrographs of the high temperature ends of the tilt-tubes showing attack by the bismuth. Figures 19 and 20 are photomicrographs of the deposit in the cold end showing the microstructure.

4. X-ray spectrographic analysis

In addition to characteristic lines of vanadium and bismuth, two strong lines of an unidentified substance are present in the X-ray spectrograph of the 168 hour cold end

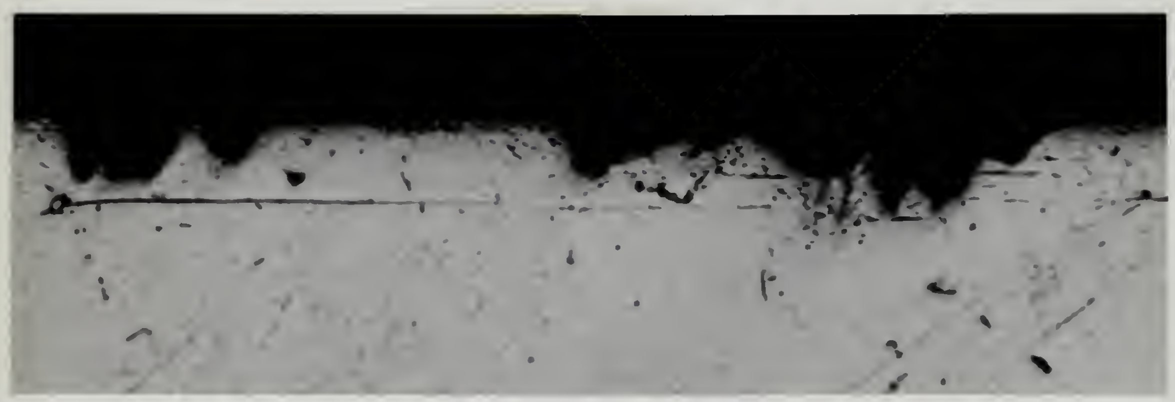
Duration of test (hours)	Temperature of hot end (degrees C)	Temperature of cold end (degrees C)	Free Liquid Metal Remaining (per cent)	Chemical Analysis of cold end deposit
1	900	300	98	no deposit
168	900	390	35	3.06% vanadium 96.94% bismuth
336	900	320	8	6.25% vanadium 93.75% bismuth
500	900	300	2	4.65% vanadium 93.35% bismuth

TABLE III Data on Mass Transfer Test

deposit. A vanadium-bismuth alloy is clearly indicated. Since extra lines introduced by this alloy are so few, probable existance of a cubic phase is inferred.

(a)

Temperature : 550°C
Period : 24 hours
Agitation : mild



(b)

Temperature : 550°C
Period : 24 hours
Agitation : severe

Figure 5

Cross section of interior surface of vanadium crucibles containing bismuth. Unetched. Magnification 600X.

(a)

Temperature : 550°C
Period : 360 hours
Agitation : mild

(b)

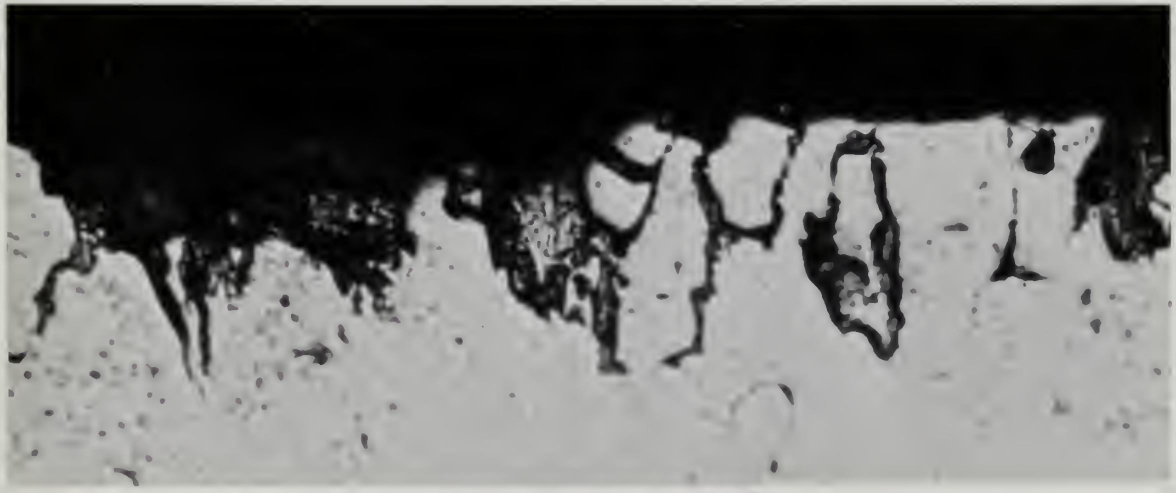
Temperature : 550°C
Period : 360 hours
Agitation : severe

Figure 6

Cross section of interior surface of vanadium crucibles containing bismuth. Unetched. Magnification 600X.

(a)

Temperature : 630°C
Period : 96 hours
Agitation : mild

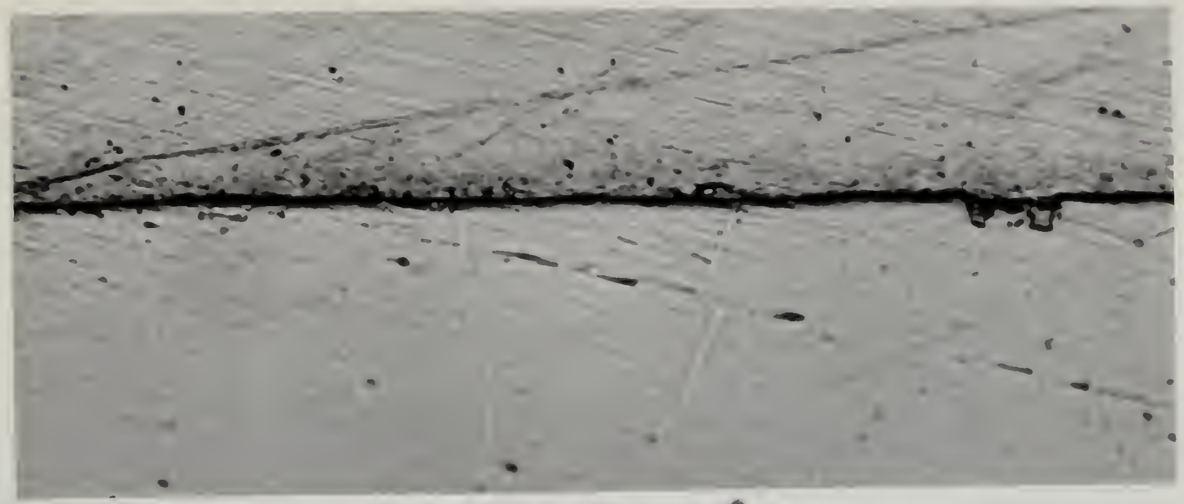


(b)

Temperature : 630°C
Period : 96 hours
Agitation : severe

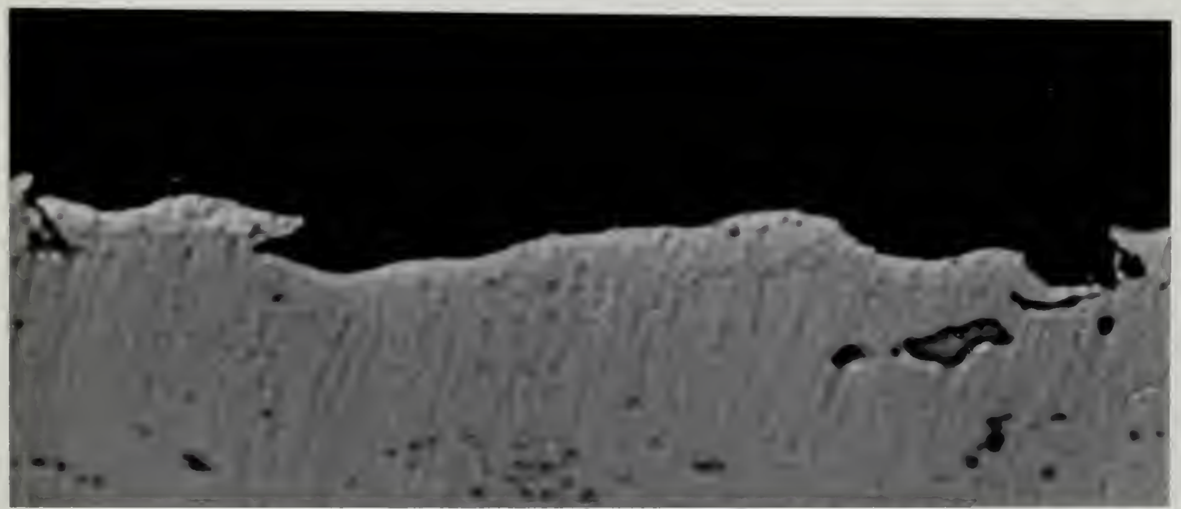
Figure 7

Cross section of interior surface of vanadium crucibles containing bismuth. Unetched. Magnification 500X.



(a)

Temperature : 720°C
Period : 96 hours
Agitation : mild



(b)

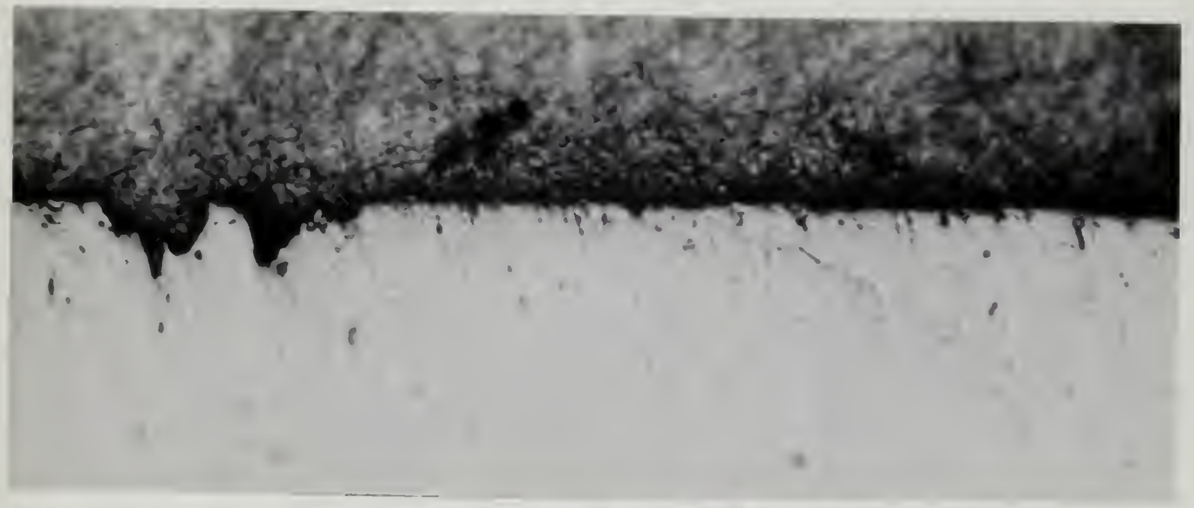
Temperature : 720°C
Period : 96 hours
Agitation : severe

Figure 5

Cross section of interior surface of vanadium crucibles containing bismuth. Unetched. Magnification 600X.

(a)

Temperature : 800°C
Period : 24 hours
Agitation : mild



(b)

Temperature : 800°C
Period : 24 hours
Agitation : severe

Figure 9

Cross section of interior surface of vanadium crucibles containing bismuth. Unetched. Magnification 600X.

(a)

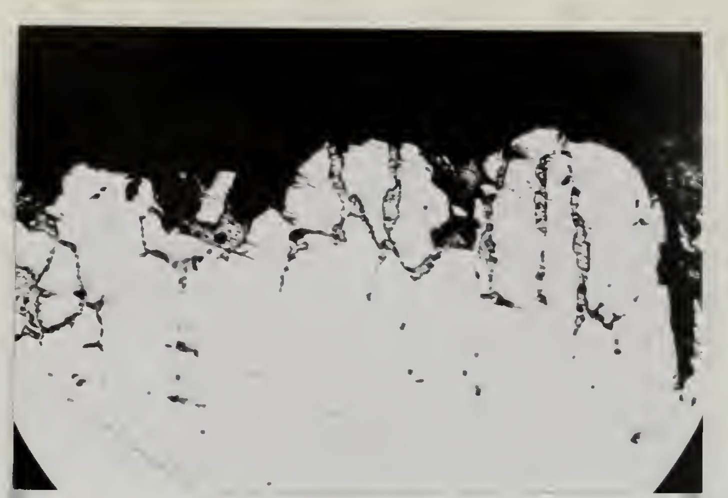
Temperature : 800°C
Period : 338 hours
Agitation : mild

(b)

Temperature : 800°C
Period : 338 hours
Agitation : intermediate (substituted for severely agitated specimen, which failed)

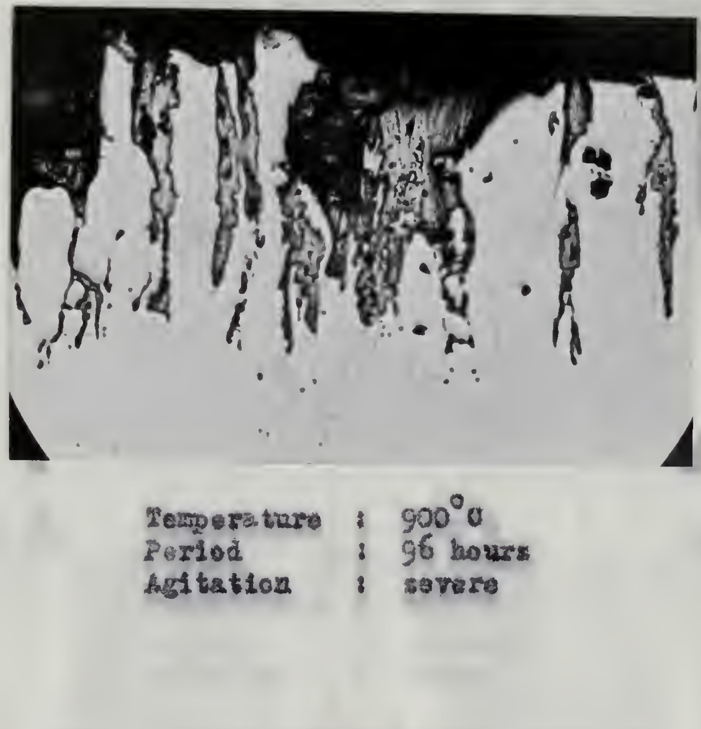
Figure 10

Cross section of interior surface of vanadium crucibles containing bismuth. Unetched. Magnification 600X.



(a)

Temperature : 900°C
Period : 96 hours
Agitation : mild

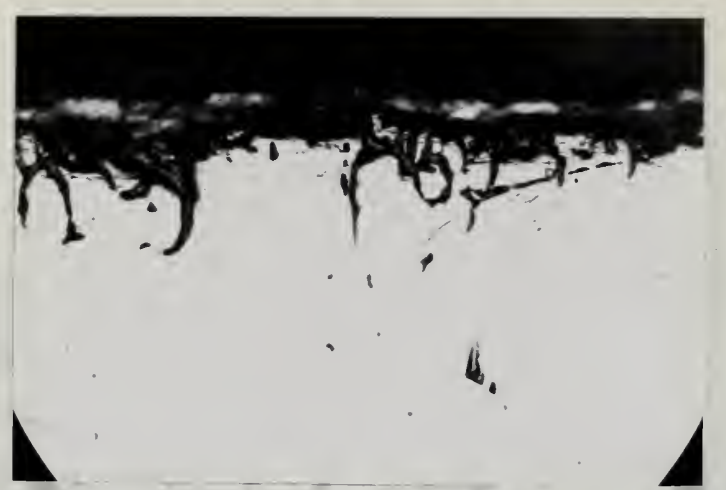


(b)

Temperature : 900°C
Period : 96 hours
Agitation : severe

Figure 11

Cross section of interior surface of vanadium crucibles containing bismuth. Unetched. Magnification 300X.



(a)

Temperature : 1000°C
Period : 24 hours
Agitation : mild

(b)

Temperature : 1000°C
Period : 24 hours
Agitation : severe

Figure 12

Cross section of interior surface of vanadium crucibles containing bismuth. Unetched. Magnification 300X.

(a)

Temperature : 1000°C
Period : 72 hours
Agitation : mild

Note: 72-hour specimen substituted for
144-hour specimen which failed under test.

(b)

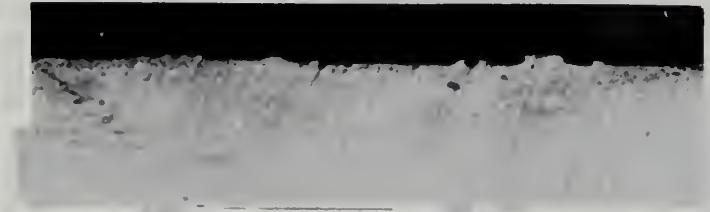
Temperature : 1000°C
Period : 72 hours
Agitation : severe

Note: 72-hour specimen substituted for
144-hour specimen which failed under test.

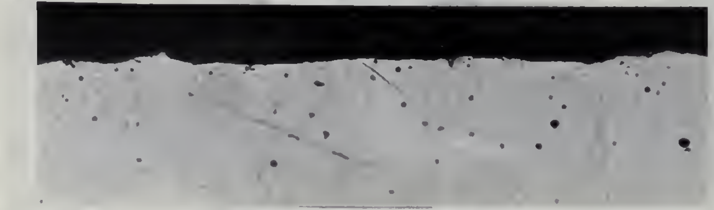
Figure 13

Cross section of interior surface of vanadium crucibles containing bismuth. Unetched. Magnification 300X.

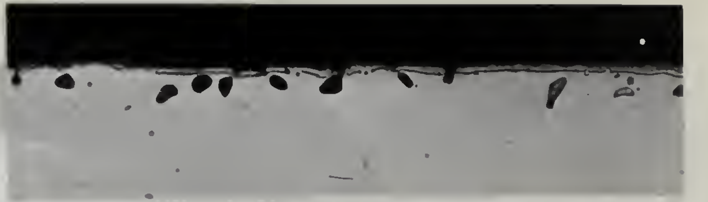
(a) Temperature : 550°C
Period : 24 hours
Agitation : severe



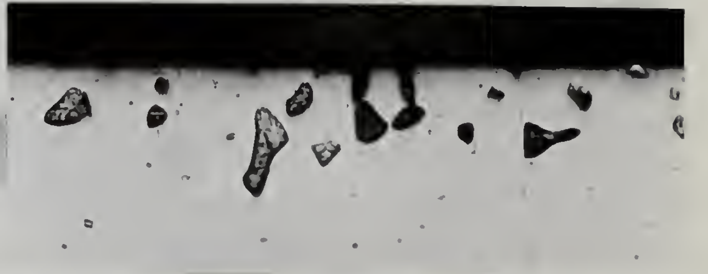
(b) Temperature : 800°C
Period : 24 hours
Agitation : severe



(c) Temperature : 800°C
Period : 338 hours
Agitation : severe



(d) Temperature : 1000°C
Period : 24 hours
Agitation : severe

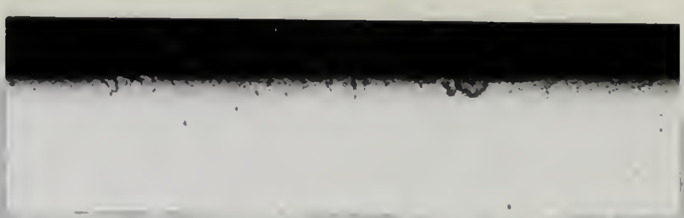


(e) Temperature : 1000°C
Period : 144 hours
Agitation : severe

Figure 14

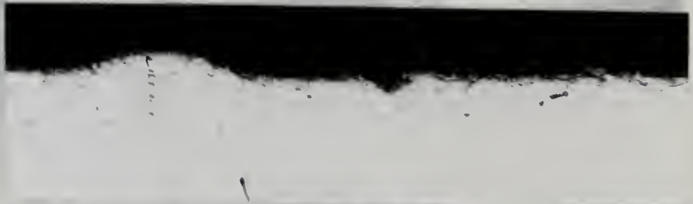
Cross section of interior surface of steel (Croloy 5 Si) crucibles containing bismuth. Unetched. Magnification 300X.

(a) Temperature : 550°C
Period : 24 hours
Agitation : intermediate



(c) Temperature : 600°C
Period : 24 hours
Agitation : intermediate

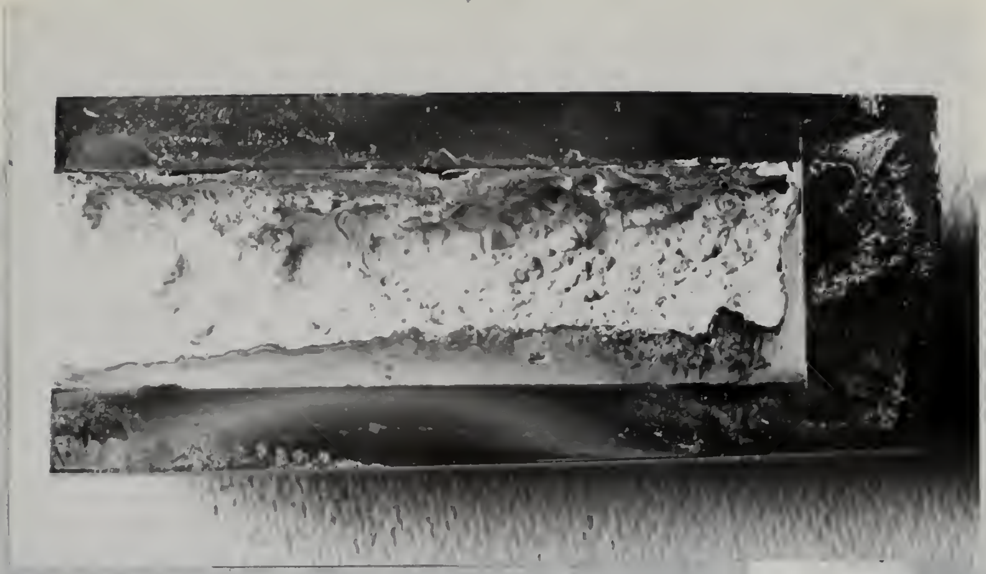
(b) Temperature : 550°C
Period : 360 hours
Agitation : intermediate



(d) Temperature : 600°C
Period : 338 hours
Agitation : intermediate

Figure 15

Cross section of interior surfaces of vanadium crucibles containing sodium. Unetched. Magnification 300X.



(a) Cycling Rate: 5 minutes. Duration of Test: 168 hours.
Unetched. Magnification : 4X

(b) Cycling Rate: 5 minutes. Duration of Test: 336 hours.
Unetched. Magnification : 6X

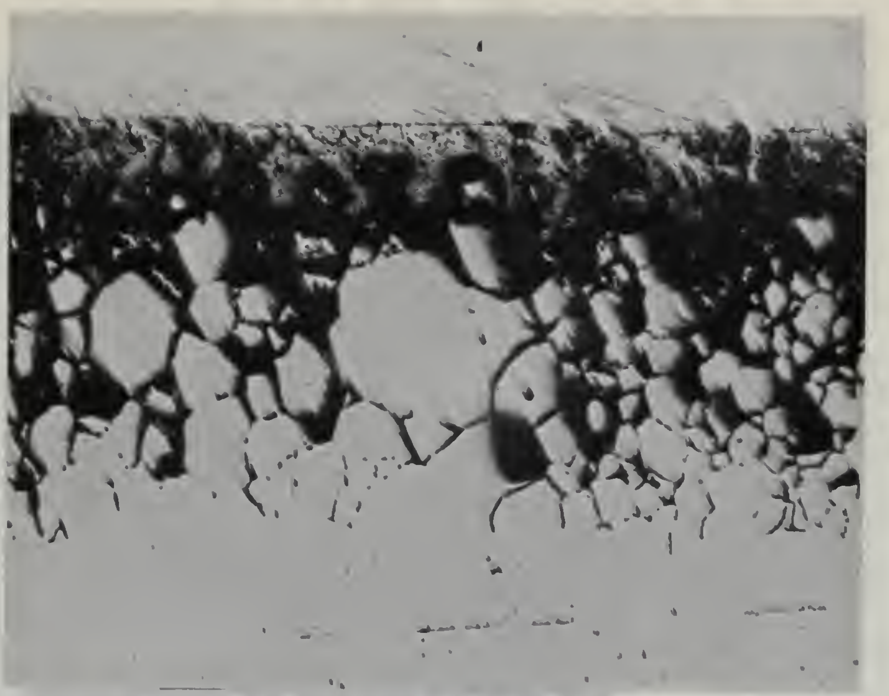
Figure 16

Macrophotograph of cross sections of low temperature ends of mass transfer tilt tubes.

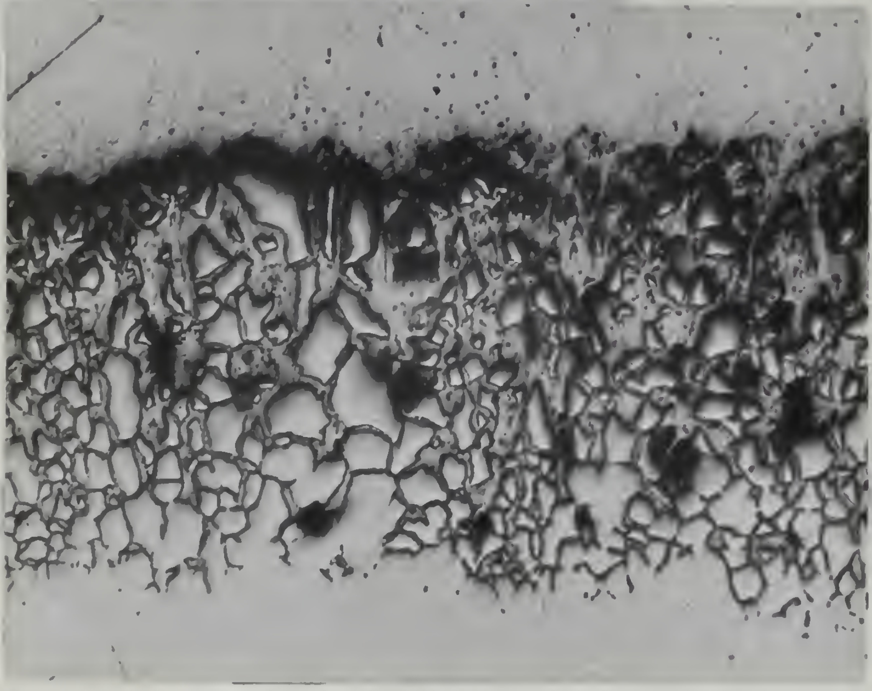
(a) one hour (b) 168 hours (c) 336 hours

Figure 17

Macrophotographs of high temperature ends of tilt tubes cycled for 1, 168, and 336 hours. Unetched. Magnification: 3X.



(a) Duration of Test: 168 hours.



(b) Duration of Test: 336 hours.

Figure 18

Cross section of high temperature ends of mass-transfer tilt tubes. Unetched. Magnification: 150X.

Figure 19

Photomicrograph of deposit in low temperature end of tilt tube. Duration of Test: 168 hours. Unetched. Magnification: 90X.

Material deposited
in
cold end of tube.

Entrapped
Liquid
Metal

Vanadium

Figure 20

Photomicrograph of deposit in low temperature end of tilt tube.
Duration of Test: 336 hours. Unetched. Magnification: 90X

V. DISCUSSION OF RESULTS

A. Corrosion tests

1. Solubility tests

The solubility of iron in molten bismuth was low up to about 750° C above which it is seen to increase rapidly as shown in Figure 4. The vanadium solubility curve, within the temperature range tested, exhibits much greater linearity, not having a particular temperature above which corrosion accelerates rapidly. Neither, on the other hand, does it have a temperature region in which its solubility characteristics may be considered good. The almost linear increase of solubility with temperature establishes conditions favorable to thermal gradient mass transport at any selected set of operating temperatures. Solubility of vanadium in bismuth was greater than that of iron in bismuth at all temperatures tested.

2. Corrosion and corrosion-erosion

Marked difference in surface attack at low temperatures are evident (Figures 5 and 6) between specimens which had gentle agitation and those which had severe agitation. At first, attack increases as temperature is increased, showing definite intergranular attack at 630° C (Figure 7). However, intergranular attack diminishes at the temperatures at which intermetallic film formation becomes manifest. At 720° C, one of the specimens shows film formation with adherent bismuth. The other did not form a film and

was corroded (figure 8). At 800° C intermetallic film formation was fairly consistent and the surfaces were only slightly damaged. At 900° C and above, intense intergranular attack again becomes evident, reaching proportions of 500 mils per year at 1000° C. Measurements of penetration at these temperatures is rather arbitrary since damage is often so advanced as to make the location of the original surface level a matter of conjecture. Attack, at 1000° C, appears more severe for specimens agitated mildly than for highly agitated specimens. This may be caused by mechanical breaking away, under severe agitation, of grains whose boundaries have been chemically attacked, giving the appearance of less penetration.

B. Corrosion-erosion of vanadium by sodium

Erosion at low temperature is again evident though generally to a lesser degree than that caused by bismuth. Attack was worse at 550° C than at 800° C, amounting to 197,000 parts per million at 550° in 360 hours. The surfaces at 800° are characterized by bumps or protuberances. These bumps are believed to be localized points resistive to corrosion attack, possibly due to impurities present. Attack is believed to be a general surface attack, leaving the resistive points salient from the lowered surface.

Though this set of tests are of a rough and posibly inconclusive nature, it appears that vanadium does not hold much promise as a container material for molten sodium.

C. Thermal gradient mass transport tests

Evidence of mass transfer under the extreme conditions imposed in this test is incontrovertible. After completion of the tests it was evident that more bismuth should have been introduced into the mass-transfer tubes. Upon sectioning the tube after a test period of 336 hours, almost all bismuth was depleted (see Figure 17). A large proportion of it had apparently gone into a stalagmitic growth in the center of the tube adhering to the lower side and to the end. The growth was brittle and of a very fragile nature and was broken in attempting to prepare it for photographing. A considerable portion of the bismuth had apparently gone into a gold-colored layer adhering to the walls of the tube beginning at about the center of the tube and becoming quite thick at the high temperature end. The appearance of the film may be noted in Figure 17.

Corrosion attack of the prepared surface at the hot end of the mass transfer tubes was intergranular in nature and was so severe for the cycling periods of 168 and 336 hours that the lowest power available in the microscope barely included the full depth of penetration in its field. There was no apparent attack in the tube operated for only one hour. In the 500 hour test, pentration was visible to the naked eye (about 1.5 Millimeters).

VI CONCLUSIONS

A. Commercial quality high-ductility vanadium is not a suitable container material for high purity molten bismuth because of the following demonstrated properties:

1. low resistance to mechanical attack at temperatures between 550 and 700°C
2. High solubility in molten bismuth
3. severe inter-granular corrosion at high temperatures
4. susceptibility to thermal gradient mass transport. (Convection currents of bismuth in a vanadium circuit may be expected to produce plugging in the cold portion of the loop and severe attack in the hot portion).

B. Commercial quality vanadium shows little promise as a container material for molten sodium.

VII RECOMMENDATIONS

The light weight, abundance, high strength-weight ratio, high-temperature strength, and reasonable fabrication characteristics (5, 6), coupled with a small neutron absorption cross-section (7) make vanadium a highly desirable material for use in nuclear reactors, (particularly in fast reactors). In order to evaluate its feasability for use in a fast homogenous reactor further research, using the techniques of the foregoing experiments, could be continued along three general lines:

1. evaluation of vanadium alloys, such as a vanadium-2½% zirconium alloy (8), as container materials for bismuth
2. evaluation of pure vanadium and vanadium alloys as container materials for other easily-liquified metals which have low neutron absorption cross sections, such as gallium, lead, lithium, thallium, tin, and low-melting eutectics of lead-bismuth (3)
3. evaluation of inhibitors, such as zirconium and magnesium, in a vanadium bismuth system to eliminate the selective attack at the grain boundaries and reduce the rate of solution.

Appendix A

Furnace Design and Construction

Design parameters for the furnace were imposed by an unusual set of circumstances dictated by the testing procedure decided upon. Because of the shocks and mechanical stresses to which the furnace would be subjected, it was imperative that the windings be exceptionally rugged. The physical size of the furnace had to be fairly small so that the teeter-board dimensions would be reasonable, yet the isothermal region within the furnace had to be relatively large. The latter criterion necessitated multiple windings with the individual windings controllable independently. In the presence of a constant-power-per-turn winding, temperature in this type of furnace usually distributes itself along a cosine curve on the longitudinal axis because of the maximum heat loss to the ends of the core. To compensate for this, windings were designed with maximum power per turn at the ends and minimum at the center. For rigidity and high temperature strength, a very heavy wire, twelve gauge was decided upon. The large diameter and low resistance of the wire enforced the use of transformers to provide low-voltage high-power sources. Five zones of controllable heating were decided upon in order to insure that constant temperature could be maintained over a large portion of the furnace length. Since the transformers provided isolation of the power sources, adjacent windings shared lead-in wires and internal wiring was kept to a minimum. The twelve guage wire was wound on a three inch diameter Alundum refractory tube, 18 inches in length , grooved

6 turns to the inch. The end windings, consisting of 14 turns each, were energized by 24 volts (nominal voltage). These windings extended no further than the end plugs of thecore, and were intended to heat the plugs and prevent heat loss and consequent temperature drop at the ends. The actual interior portion of the furnace was heated by three windings of 22, 34, and 22 turns respectively, energized by voltages (nominal) of 30, 36, and 30 volts. The primary of each isolation and step-down transformer was energized by a Variac variable voltage transformer by which the output voltage could be varied from 0 to 117 % of nominal voltage. A chromel-alumel thermocouple was placed directly adjacent to the center of the winding and used for operation of a Leeds and Northrup Micromax controller. The controller operated a holding type relay (to prevent relay chatter) which, during demand periods, shorted out a large Variac used as a variable inductance in series with all Variacs controlling furnace region isolation transformers. During non-demand periods the voltage to the isolation transformers was reduced by the drop across the series inductance. This method of control was the only one of the three tried which did not result in excessive relay contact arcing due to inductive effects of the five isolation transformers in parallel. A circuit diagram of the furnace winding and control circuit is shown in Figure 21.

Leads from the individual windings were welded to the common points of the windings and consisted of double lengths of 12 gauge wire spot welded together every two inches. Ends of the double wires were welded

to studs which passed through the transite furnace supports and were used as connection points. Using equal power in all windings (not equal power per turn) the furnace temperature between end plugs was within ten per cent of the center set temperature (maintained by controller) upon first heating. By adjustment of voltage to the individual zones, temperatures within the furnace could be maintained to within plus or minus 1/2% at all points.

The frame for the furnace teeter-board (Figure 1) was made using four-inch channel beams and was supported, at first, in brass sleeve bearings. The weight of the furnace caused binding of the sleeve bearings. This interfered with the bouncing action intended to supply severe agitation to the end-mounted specimens. Large ball bearings were substituted for the sleeve bearings and no further bearing difficulty was experienced.

For the corrosion tests the microswitch and operating cam shown Figure 1 were not used. The motor speed controller was set to bounce the furnace on the springs once every 5 seconds.

For the mass transfer tests the bouncing action of the furnace teeter-board was damped out, and the oscillation period reduced to a ten minute cycle.

Oscillation frequency was reduced by periodic interruption of power to the motor by an electronic interval timer. The circuit diagram of the interval timer is shown in Figure 22. The cam operated the microswitch as the furnace teeter-board went through horizontal dead

center. The power was then cut off to the motor, but inertia kept the motor going for a few more turns. From this point on the furnace dropped freely onto a damped stop. At the end of the timed interval the intermittent lift action of the operating mechanism allowed the motor to build up to speed under no load conditions.

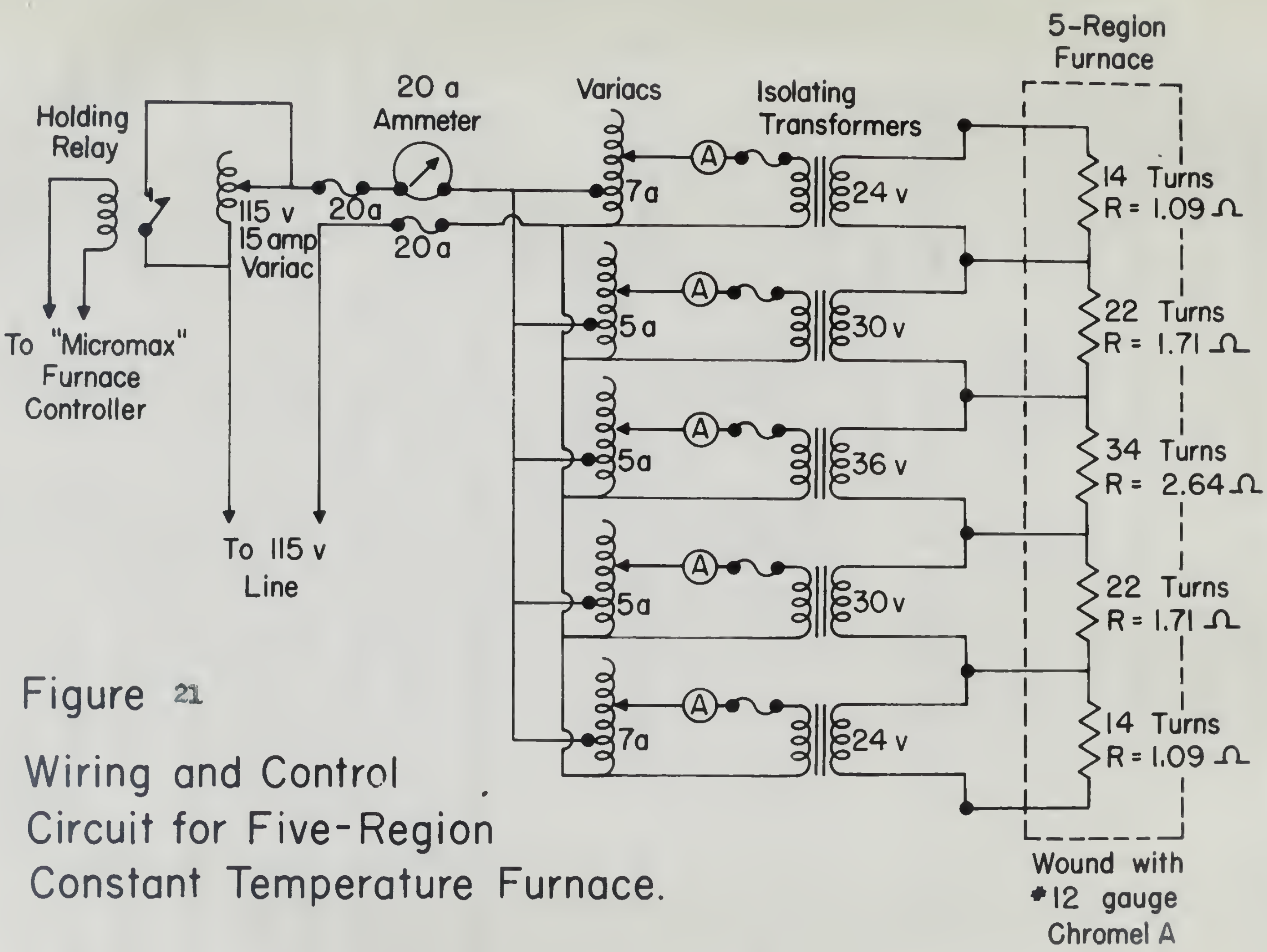


Figure 21

Wiring and Control Circuit for Five-Region Constant Temperature Furnace.

APPENDIX B

Crucible Construction, Loading, and Welding

Specifications for the starting materials are as follows:

1. vanadium metal (Electro-Metallurgical Co.)

form: chips, 1/4 inch by 12 mesh

analysis :

carbon	0.05%
oxygen	0.056%
hydrogen	0.01 %
nitrogen	0.06 %

2. bismuth metal (Mallinckrodt Chemical Works)

form : granulated

analysis :

arsenic	0.008 %
copper	0.005 %
iron	0.00 %
silver	0.005 %
zinc	0.00 %

3. sodium metal (Mallinckrodt Chemical Works)

form : solid billets

analysis : not available

The vanadium metal chips were cold compacted in a steel can at 32,000 pounds per square inch. The can was then evacuated, sealed off and extruded at 1950° F through a 0.7 inch die, the extrusion constant constant being 64,000 pounds per square inch. The first extrusion was largely unusable because of insufficient back pressure, resulting in cracks and fissures in the rod. Subsequent extrusions were made with a nickel plug at the front of the extrusion billet. The nickel, not being as soft as the steel at 1950° F greatly increased the initial back pressure and resulted in an extrusion of much better quality. The rods were swaged lightly for straightening, then turned down on a lathe to the final outer diameter, thereby removing the steel jacket.

Discarding the ends of these rods (which were contaminated with nickel at the front and with steel at the back) left a solid vanadium rod. Sectioning of the rods into crucible lengths was followed by the drilling out of each section to proper depth. Interior surfaces were polished with fine emery cloth. Capsfor the crucibles were prepared from the same material, the inner surface being given a metallographic polish. Dimensions of the assembled crucible are included in Figure 2. Just prior to charging these crucibles with bismuth metal, they were thoroughly cleaned by pickling in concentrated nitric acid.

It was felt that the metallic bismuth, despite its high purity, should not be placed directly into the vanadium crucibles due to the large surface area in its granulated form and the existence, at room temperature, of an oxide film on bismuth. The surface area was greatly reduced by melting down the granules in a graphite crucible (in air) and pipetting unoxidised metal from below the surface. The metal was drawn up into a 1/4 inch (inner diameter) Pyrex tube and allowed to freeze, after which the glass was broken away and the bismuth rods cut into 4.5 gram lengths. The crucibles were charged with these just before welding.
In the case of the sodium, efforts to clean up the material were limited to slicing away the contaminated surface with a spatula thereafter reducing the period of exposure in air to a minimum.

Following charging with either bismuth or sodium, the crucible cap was inserted, and the assembly placed in the vacuum arc melting unit (Figure 22). The water-cooled copper crucible shown in Figure 22 was replaced by a solid copper cylinder which was

honey-combed to contain (with snug mechanical fit) seven of the crucibles at one end, and flowing coolant (water) at the other. This fixture successfully kept the temperature of the crucibles to a minimum in those regions surrounding the contained bismuth or sodium, although welding of the capsules containing sodium proved troublesome due to vaporization of the sodium when the arc was maintained for more than a few seconds at a time. Previous to welding, the arc furnace was evacuated to an absolute pressure of less than one micron, then filled to a pressure of 1.1 atmosphere with argon bled into the arc melting cylinder through a purifying train. A tungsten electrode was utilized in the welding process.

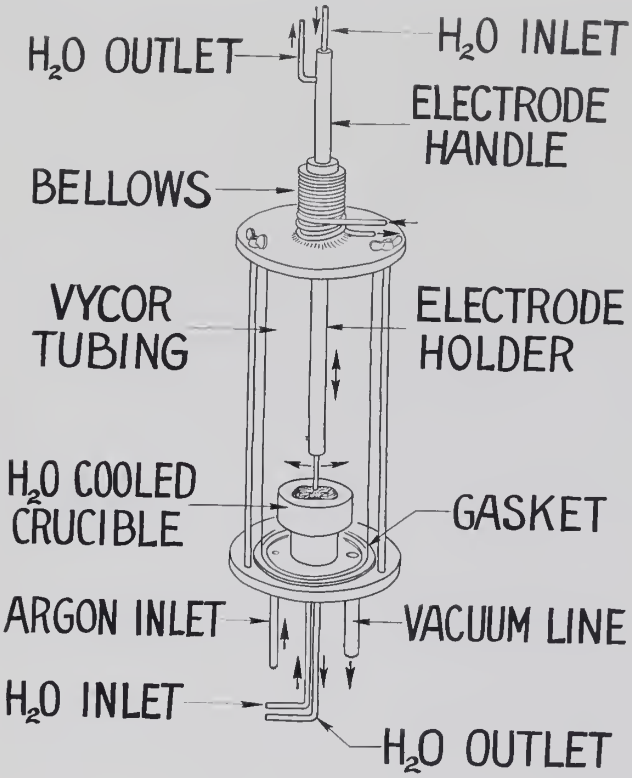


Figure 22

Drawing of arc furnace used for welding of specimens.

APPENDIX C

Chemical Analyses

1. Determination of vanadium in bismuth

a. Preparation for chemical analysis

Upon removal from the furnace the crucibles were placed vertically, prepared surface up, without cooling, so that particles eroded from the crucible would have a chance to settle. The crucibles, in the same position, were then quenched in cold water. The crucible material and approximately 1/32 inch of the outer surface of the bismuth slug were turned off in a lathe and the slug cut off 1/8 inch from the point where it adhered to the bottom of the crucible. The slug was then immersed in nitric acid and reduced to approximately 1/8 inch in diameter by 1/2 inch length. It was intended by this means to eliminate all vanadium from the bismuth except that which had been taken into solution during the test period. As a cross-check of the success of the process in removing all but dissolved material the remaining slugs were divided into two parts which were checked separately. If the two parts did not match within about 30% the two solutions made up from the two sections of the slug were rechecked. This accounts for the extra entries for some of the specimens in Table II.

b. Chemical procedure

(1) One fourth gram (.25 g) of each of the parts of the bismuth slug were taken into solution with 30 drops of concentrated nitric acid

(2) washed down

(3) ten milliliters (10 ml) concentrated sulfuric acid added

(4) washed down and fumed to sulfuric

(5) step (4) repeated twice

(6) cooled, diluted to 50 cc with distilled water

(7) one gram of ammonium cloride added

(8) solution transferred to 100 ml volumetric flask

(9) 5 drops of hydrogen peroxide added

(10) diluted to 100 ml, mixed, and color allowed 30 minutes to develop

(11) color compared with standard sample in Beckman spectrophotometer at a wavelength of 450 millimicrons.

2. Determination of Vanadium in Sodium

a. Preparation for chemical analysis

Upon removal from the furnace, crucibles of vanadium containing sodium were quenched in cold water. The tops of the crucibles were cut off using a dry hack saw and all filings and cuttings blown away ith compressed air. The entire crucible was then immersed in distilled ater for removal of the sodium. When the reaction had subsided, the nterior of the crucible was flushed of remaining particles with stilled water which was added to the immersion water. The crucible ntents were then in the form of solid vanadium particles and sodium droxide in water.

b. Chemical Procedure

(1) 15 ml sulfuric acid and 5 ml nitric acid added

(2) fumed to sulfuric acid

(3) while fuming, nitric acid added dropwise

(4) fuming and addition of nitric acid continued until all vanadium is in solution

(5) cooled and transferred to 100 ml volumetric flask

(6) diluted to mark and mixed

(7) aliquots were taken to contain not more than 200 micrograms of vanadium

(8) five drops of hydrogen peroxide added

(9) diluted to mark with 10% sulfuric acid, color allowed 20 minutes to develop

(10) color measured in Beckman spectrophotometer at wavelength of 450 millimicrons

(11) 20 ml aliquots from the original solution taken (for sodium measurement)

(12) 10 mg iron added and an ammonium precipitation made

(13) to the filtrate from the ammonium precipitate, sulfuric acid was added until the solution was just acid to litmus

(14) 3 ml hydrocloric acid and 2 ml nitric acid added

(15) solution evaporated to dryness

(16) cooled, salts dissolved in distilled water, transferred to tared platinum dish and again evaporated to dryness

(17) salts were taken up in water and a drop of ammonium hydroxide added

(18) evaporated to salts and the salts heated until fumes cease and fusion begins

(19) cooled and weighed

(20) sodium content calculated from sodium sulfate present, and parts of vanadium per million parts of sodium determined

3. Determination of Iron in Bismuth

a. Preparation for chemical analysis

Upon removal from the furnace, steel crucibles were held vertically for settling of particles, quenched, machined, and cleaned in a manner identical to that described in part 1 of Appendix C.

b. Chemical procedure

(1) a weighed sample of the bismuth was placed in a 250 ml beaker and enough nitric acid added to take samples into solution

(2) evaporated nearly to dryness

(3) 5 ml hydrochloric acid added and taken to dryness

(4) step (3) repeated

(5) 10 ml hydrocloric acid added

(6) ammonium chloride added in ratio 1 g for every 0.1 g of sample

(7) diluted with distilled water to 100 ml

(8) heated gently until all ammonium chloride was taken into solution

(9) transferred to volumetric flask, diluted to mark, and aliquots taken equivalent to about 0.1 g samples

(10) aliquot diluted to approximately 40 ml

(11) added 3 ml hydroquinone solution (1%)

(12) added 6 ml of 1-10 ortho-phenanthroline (0.25 %)

(13) 6 ml ammonium citrate (molar solution) added, stirring between additions

(14) A crystalline precipitate formed

(15) Added one-to-one ammonium hydroxide

(16) Adjusted pH to 3.5

(17) Solutions were transferred to 100 ml volumetric flasks, diluted to mark, and mixed

(18) Allowed to stand 20 hours for full color development

(19) Optical density read in Beckman spectrophotometer at wavelength of 513 millimicrons.

Reagent blank treated in same manner was used for correction.

APPENDIX D

Construction, Loading and Welding of Mass Transfer Tubes

Starting with an Electromet cast ingot of vanadium, an attempt was made to extrude vanadium tubing over a 3/4 inch mandrel. The mandrel failed in the extrusion, being "sucked out" with the metal. The remaining tube broke apart in the first pass through a swaging machine. Hardness of this material was measured as approximately R_A 46. It was postulated that the hardness might have been caused by hydrogen absorption evolved in the process of "pickling" the iron from the extruded tube. After vacuum annealing for three days at 900° C the hardness was approximately R_A 40. The idea of extruding tubing with available equipment in the in the time remaining was abandoned.

Vanadium rods were extruded as before. (see Appendix C) The iron jacket was turned off in a lathe, and the rods cut into eight inch lengths. Holes were bored through the center of the rods and reamed smooth.

Caps, with metallographically polished inner surfaces were welded into the ends in an argon atmosphere using the apparatus described in Appendix A and shown in Figure 22. For welding the long tubes, the only modification to the welding apparatus was the substitution of a high glass cylinder in place of the one normally used. The tubes were charged with ten grams of bismuth purified, as before, by pipetting from below the surface of a pool of molten bismuth and freezing the pipetted material.

All precautions taken to prevent contamination of the crucibles described in Appendix B, such as pickling of tubes just prior to charging with bismuth, purification argon in the arc-melting unit, etc., were duplicated in preparation of the mass transfer tubes.

APPENDIX E

Electronic Interval Timer

Calculations based on best estimates of heat conduction coefficients and constants of emissivity indicated that steady state temperatures would just be approached in the low temperature end of a tilt-tube five minutes after the bismuth came from the hot end at 900° C. The calculations were based on a full ten grams of contained metal and did not take into account depletion of the bismuth, nor was this depletion anticipated to the extent to which it occurred.

In order that the furnace remain in a tilted position for a five minute period, it was necessary to cut off power to the motor when the furnace teeter-board reached an end position. This was accomplished by attaching a cam to the furnace teeter-board which operated a single-pole double-throw switch instantaneously on and off again while the teeter-board was going through horizontal dead center. An electronic interval timer was operated by this switch. While the microswitch was operated, it charged a 15 microfarad oil-filled paper capacitor to 220 volts from a 40 microfarad electrolytic condenser. This was done so that a large charge could be put into the 15 microfarad capacitor in the very short time that the switch was on. The electrolytic then had five minutes to recharge. A 6V6 tube, triode connected, was cut off by the voltage on the 15 microfarad capacitor for the time it took to

discharge through one of several selected resistors. During the time it took to discharge, the tube was cut off, a relay in its plate circuit disconnected power from the motor. Positive relay action was ensured by a feedback circuit which connected the grid to a positive voltage through a two megohm resistor as soon as the relay began to close. Relay chatter was experienced until the feedback loop was added.

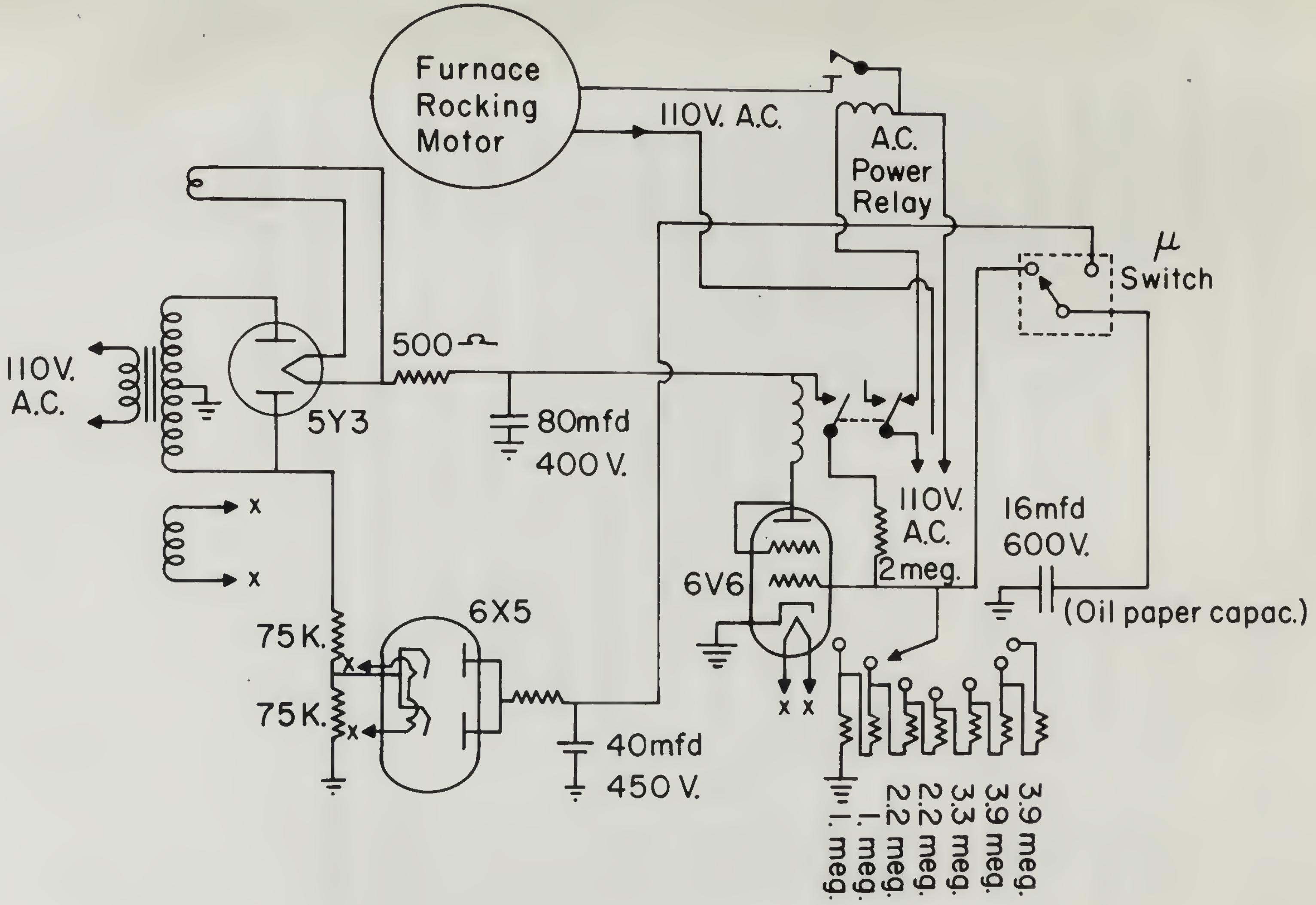


Figure 23 Electronic Interval Timer for Mass-Transfer Experiment.

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